AD-A248 810

‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖
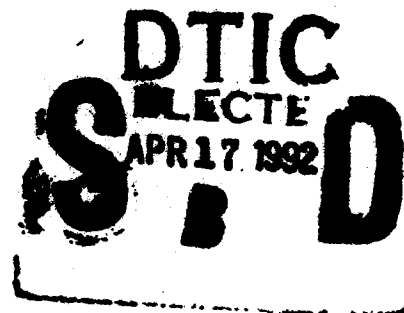
DYNAMIC MECHANICAL MATERIALS CHARACTERIZATION AND ANALYSIS USING A DYNAMIC MECHANICAL THERMAL ANALYZER

BY DR. WALTER M. MADIGOSKY
RESEARCH AND TECHNOLOGY DEPARTMENT

JANUARY 1991

DTIC
ELECTE
APR 17 1992
S B D

NAVAL SURFACE WARFARE CENTER

Dahlgren, Virginia 22448-5000 ● Silver Spring, Maryland 20903-5000

92-09716

‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖‖

92   4 15 057

DYNAMIC MECHANICAL MATERIALS CHARACTERIZATION AND ANALYSIS USING A DYNAMIC MECHANICAL THERMAL ANALYZER

BY DR. WALTER M. MADIGOSKY
RESEARCH AND TECHNOLOGY DEPARTMENT

JANUARY 1991

NAVAL SURFACE WARFARE CENTER

Dahlgren, Virginia 22448-5000 • Silver Spring, Maryland 20903-5000

FOREWORD

The purpose of this report is to document two of the dynamic viscoelastic properties (shear modulus and loss factor) of viscoelastic compounds. They are determined as a function of frequency and temperature. Applying the time-temperature superposition principle to the data, master curves are constructed and Williams-Landel-Ferry (WLF) shift constants are determined.

This work was done during FY88-89 with funds and materials provided by David Taylor Research Center (Code 1908), Bethesda, Maryland.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Materials Division

Accession For	
NTIS GRA&I	☑
DTIC TAB	☐
Unannounced	☐
Justification	

By_____
Distribution/_____

Availability Codes	
	Avail and/or
Dist	Special

A-1

CONTENTS

ILLUSTRATIONS

TABLES

INTRODUCTION

Knowledge of the dynamic mechanical properties of materials over a wide range of frequencies and temperatures is absolutely necessary in order to select materials and design systems which provide the greatest possible vibration control and noise reduction. Although there are many systems that have been designed for vibration damping and noise suppression, these systems generally have not been optimized in the strict mathematical sense. A systematic study of the problem is needed that demands not only a detailed knowledge of the dynamic mechanical properties but also a realistic mathematical model of the frequency and temperature dependence of these properties. The model parameters may then be used as input variables in a mathematical optimization computer code and, thus, both the design and the material can be chosen to guarantee optimal performance. The alternative to this is a trial and error approach which is time consuming, uncertain, and potentially expensive.

With the advent of new materials, chemicals, and processing, the need for a systematic approach is clear. Accordingly, this report describes the initial phases of a study which uses a commercial Dynamic Mechanical Thermal Analyzer (DMTA) to gather dynamic data on a number of materials as a function of temperature from the glassy to the rubbery region. Data were collected on different polymers to study the effect of polymer selection and on the polymers compounded with different loadings of a specific reinforcing filler, Durez, to study the effect of changing the hardness of the composition. The data were then analyzed according to the Williams-Landel-Ferry (WLF) equation[1] for time-temperature superposition and shifted into the frequency domain. The complex moduli were then fitted to the fractional operator model[2] which describes the moduli, storage modulus, and loss factor with a single complex equation. The simplest form of this model, used here, has only four independent parameters, all of which have a physical interpretation. Finally, an additional advantage of this approach is that these parameters, along with geometrical design parameters, may then be used as variables in a computer code that optimizes the material selection and geometry in order to achieve the maximum performance. Thus, the vibration damping or noise suppression properties of a design will be truly optimized, and no other similar design or material exists that can achieve a higher performance.

EXPERIMENTAL

Initially, two methods were used to obtain the dynamic mechanical properties as a function of temperature and frequency. The first method used a commercial DMTA from Polymer Laboratories.[3] In this method, small samples are mounted in a temperature controlled fixture, and the sample is driven at a constant frequency in either a single or double cantilevered mode. The stress necessary to achieve a given strain level and the phase angle between stress and strain is measured; the analysis of this information gives the Young's modulus and loss factor. The DMTA and its Universal Temperature Programmer (UTP) were controlled by a Hewlett-Packard Model 216 microcomputer using the software provided. Temperature control was achieved by using liquid nitrogen as a coolant and programmed heating to provide temperature scanning at low rates for good measurement accuracy. Samples to be tested were mounted using screw-on clamping bars inside the temperature enclosure and cooled to -60°C. The chamber was then momentarily opened and the clamps retightened with a torque screwdriver set to 5-10 N/cm. The DMTA was then programed to record data automatically at a ramp rate of 1 degree per minute over the temperature range of -50 to +50 degrees centigrade.

The second method of data collection used a Naval Surface Warfare Center (NAVSWC) developed resonance apparatus.[4] In this apparatus, samples were mounted vertically onto a miniature shaker while lightweight accelerometers were mounted on each end. The samples were driven with white noise and the transfer function measured as a function of frequency using a spectrum analyzer. The resulting spectrum of amplitude and phase was analyzed to yield the complex Young's modulus.

The first method had the advantage of fairly rapid and automatic data collection over a wide range of temperature but limited frequency range. The second method took longer, needed larger samples than ASTM test sheets, but provided a much larger frequency range and more accurate data over a smaller temperature interval. In the beginning, both methods were used to cross check the accuracy of the instruments and measurement procedure, especially the procedure of mounting samples in the DMTA. In addition, the greater frequency range of the resonance apparatus provided a cross check of the WLF temperature shifting procedure used in transferring the temperature data collected by the DMTA into the frequency domain. Once these procedures were established, then the bulk of the data collected (all that shown here) were collected using the DMTA and analyzed using the WLF shifting procedure.

MATERIALS AND COMPOUNDING

The materials chosen for this initial study included rubber compounds using natural, nitrile, and neoprene polymers and a commercially available urethane. In addition to the study of the effect of polymer type, the present work studied the effect of a changing the rubber modulus. In this case, the Shore "A" hardness was varied through the addition of a phenolic resin filler, Durez (made by Occidental Chemical Corp.). The formulations are shown in Table 1. The urethanes studied also varied in hardness from a Shore "A" of 60 to over 100 and are available from Conap, Inc., Olean, New York.

RESULTS

The dynamic data were recorded on the Polymer Laboratories' DMTA as a function of temperature from -50 to +50 degrees centigrade at four frequencies: 1, 3, 10, and 30 Hz. It was found that this frequency range provided sufficient data to WLF shift into the frequency domain. As mentioned above, this was checked by comparing data with that obtained by the resonance technique at higher frequencies. The advantage of the DMTA was that samples were cut directly from the ASTM sample test plates for use in the DMTA, whereas special samples had to be prepared for the resonance tester.

Figure 1 shows a typical set of data as obtained directly from the DMTA instrument. By noting the glass transition temperature, -50°C, and adjusting the two WLF shift constants, C1 and C2, where both constants are referenced to the glass transition temperature, the frequency data were converted into a single shifted curve which spans 14 decades in frequency at a reference temperature of 10°C. The results are shown in Figure 2. Note that in both the temperature and frequency presentation, the glassy and rubbery moduli and plateaus are clearly defined. Also note that the data in Figure 2 collapses into a single curve as it should if the WLF formula is applicable. Figure 3 shows similar results for four other urethane materials available from Conap which vary in hardness (Shore "A" = 60, 70, 80, 90, and 95). In each case, the glassy modulus and the glass transition temperature, Tg = 50°C, remains constant but the rubbery modulus increases as the hardness increases. Also, a single transition is observed in each of the materials. Figure 4 shows the results for another Conap material EN-4 and, in this case, a double transition is observed indicating possibly a transition in both the soft and hard segments of the urethane. Finally, the data in Figure 3 were WLF shifted and the results at 10°C are shown in Figure 5. All of the materials were found to shift satisfactorily with shift constants very near the universal constants proposed by WLF, C1 = 21 and C2 = 65, when those constants are referred to the glass transition temperature.

Similar results were found for the natural, neoprene, and nitrile rubbers; these are shown in Figures 6 through 8. In the case of these rubbers, the hardness effect was achieved through the addition of Durez. Again note that the glass transition temperature is essentially independent of the Durez as is the glassy modulus, but the rubbery modulus increases with the Durez level and the Shore hardness. Also, as in the urethane samples, the loss factor decreases as the rubbery modulus increases. The WLF shift constants were again close to the universal constants and were C1 = 19 and C2 = 51 at Tg = -60°C for the natural rubber compounds, C1 = 22 and C2 = 51 at Tg = -30°C for the nitrile rubber compounds (except compound #NBR-0 which had a Tg = -40°C) and C1 = 18 and C2 = 51 at Tg = -37°C for the neoprene rubber compounds. Finally note that the addition of Durez to the nitrile compound has a different effect on the loss factor frequency dependence in that it appears to be much broader than in the neoprene and natural compounds.

The frequency data were then fitted to the fractional derivative model which in its simplest form is:

$$E^* = Eg + (Er - Eg)/[1 + (iF/Fo)^a]$$

where E^*, Eg, and Er are the complex, glassy, and rubbery values of Young's modulus, F is the frequency, and Fo and a are two constants that are the frequency at which the imaginary part of Young's modulus is a maximum and the slope of the real part of Young's modulus at the inflection point. Fo determines where the transition occurs in frequency space, and a is a measure of the width of the transition. Thus, all four constants have physical meaning.

Each of the frequency curves were fitted to this model, and typical results for the nitrile compounds are summarized in Table 2. Note the decreasing value of a which corresponds to the increased broadening of the transition with increasing hardness.

Finally, it is clear that the four fractional derivative parameters are a realistic set of constants that maybe used as variables in a computer code which optimizes the material response to obtain a desired level of performance. Knowledge of how these constants vary with each of the ingredients in a rubber compound will make it possible to define the compound formula that has the optimal dynamic Young's modulus properties predicted by the computer code.
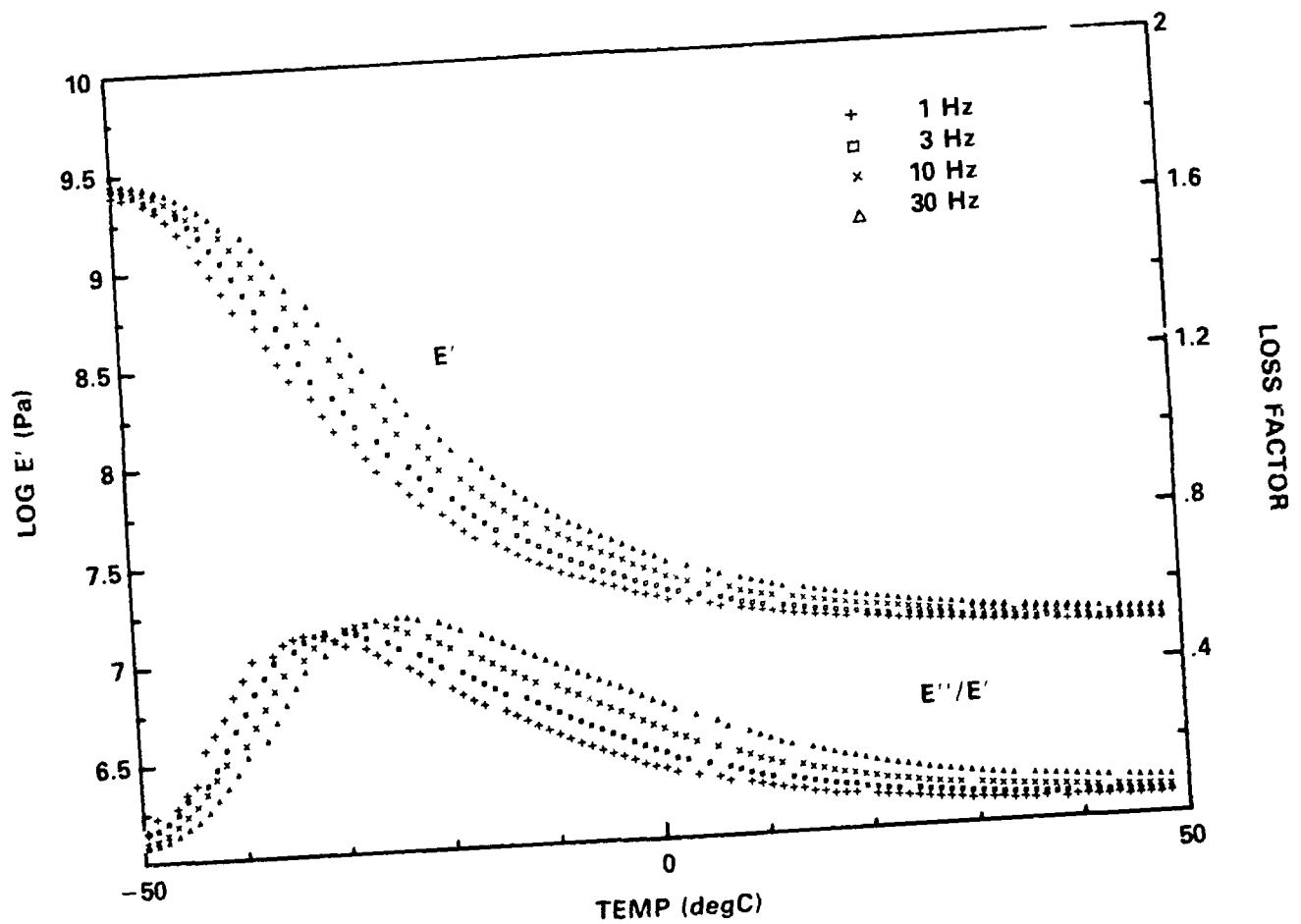
FIGURE 1. YOUNG'S MODULUS AND LOSS FACTOR FOR URETHANE POLYMER TU-700 (CONAP, INC., OLEAN, NEW YORK) AS A FUNCTION OF TEMPERATURE MEASURED BY THE DMTA
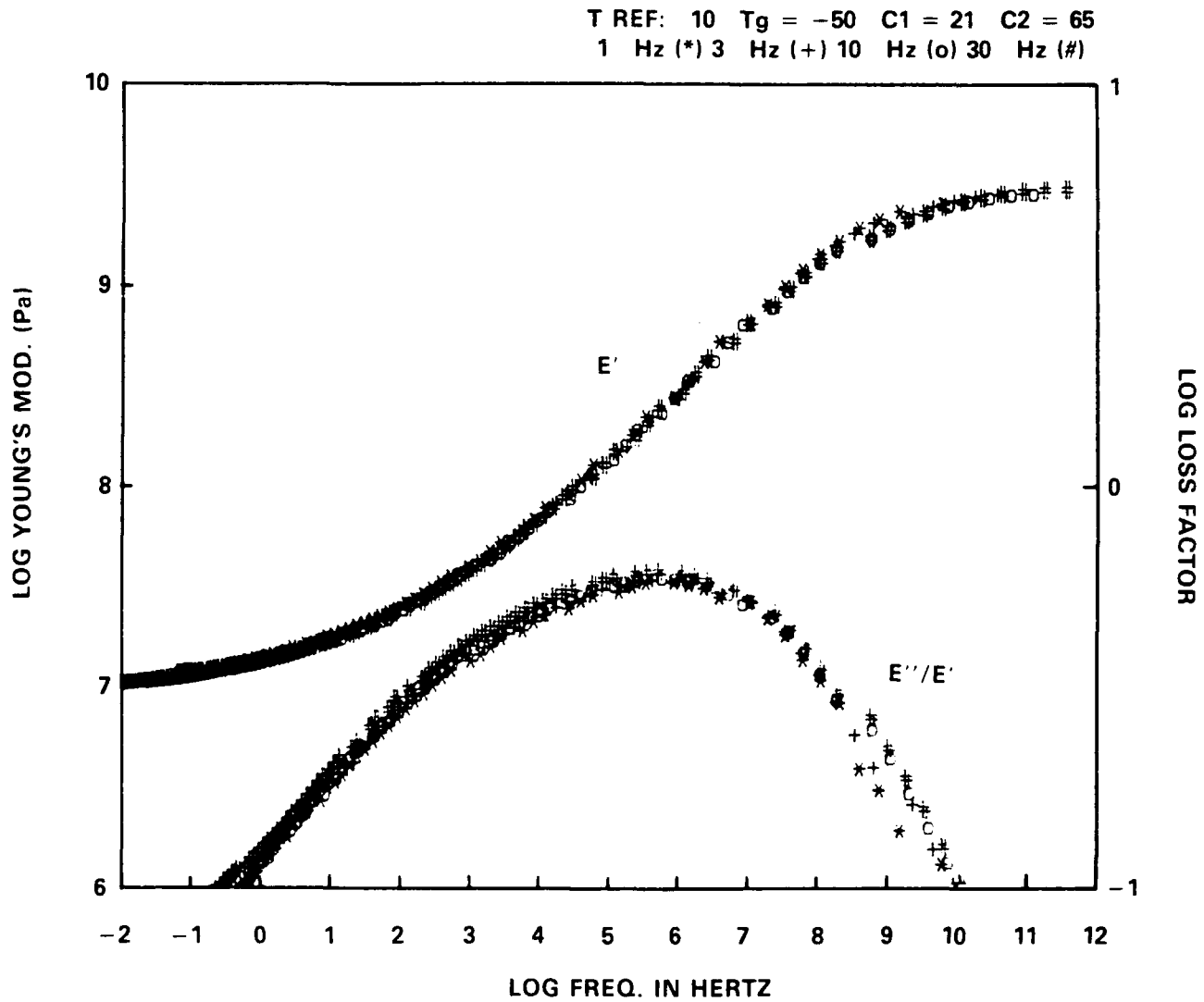
FIGURE 2. WLF SHIFTED DMTA DATA (FIGURE 1) VERSUS FREQUENCY
FOR TU-700 AT 10°C
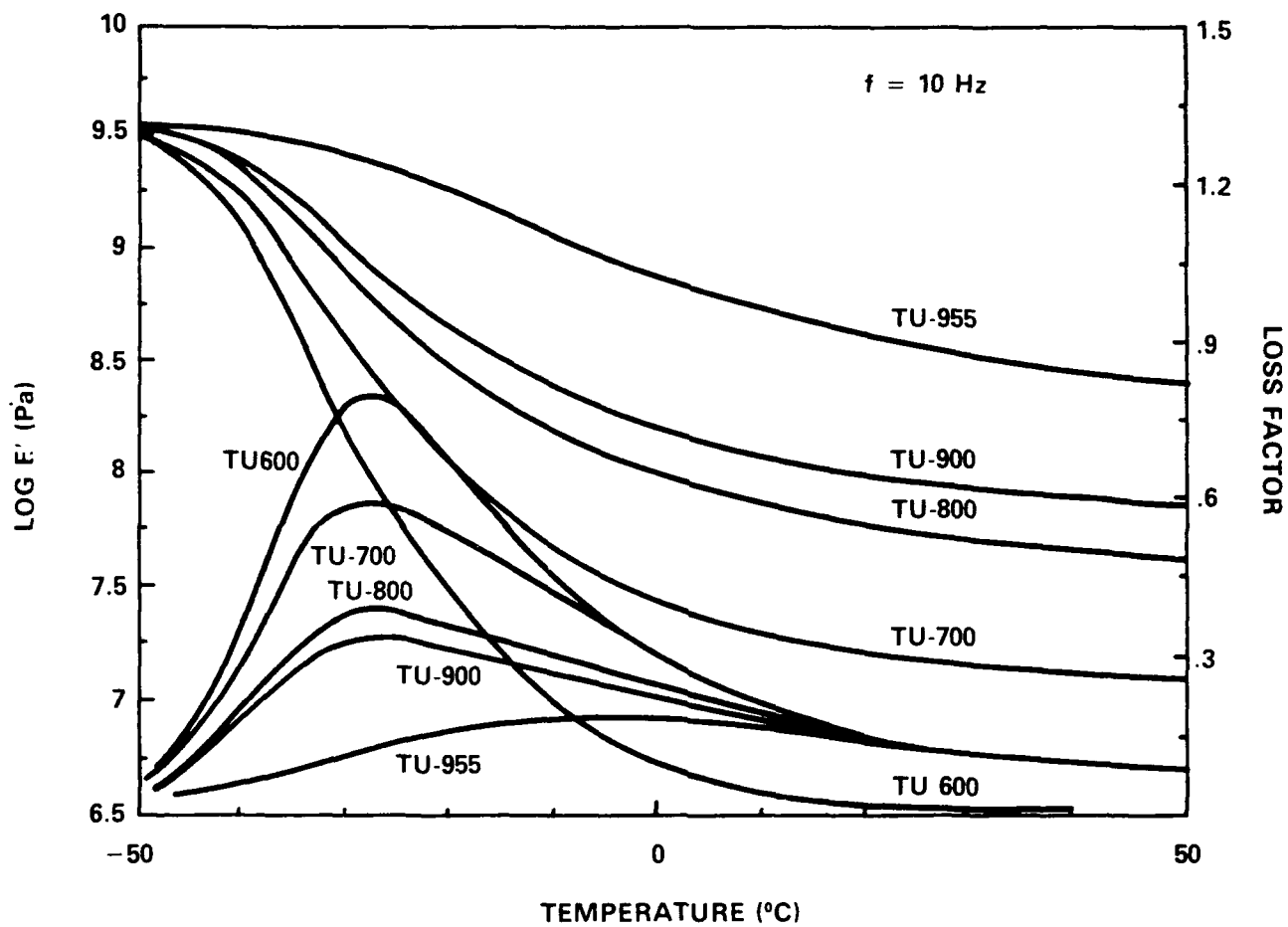
FIGURE 3. YOUNG'S MODULUS AND LOSS FACTOR FOR A SERIES OF
URETHANE POLYMERS WITH INCREASING SHORE
"A" HARDNESS
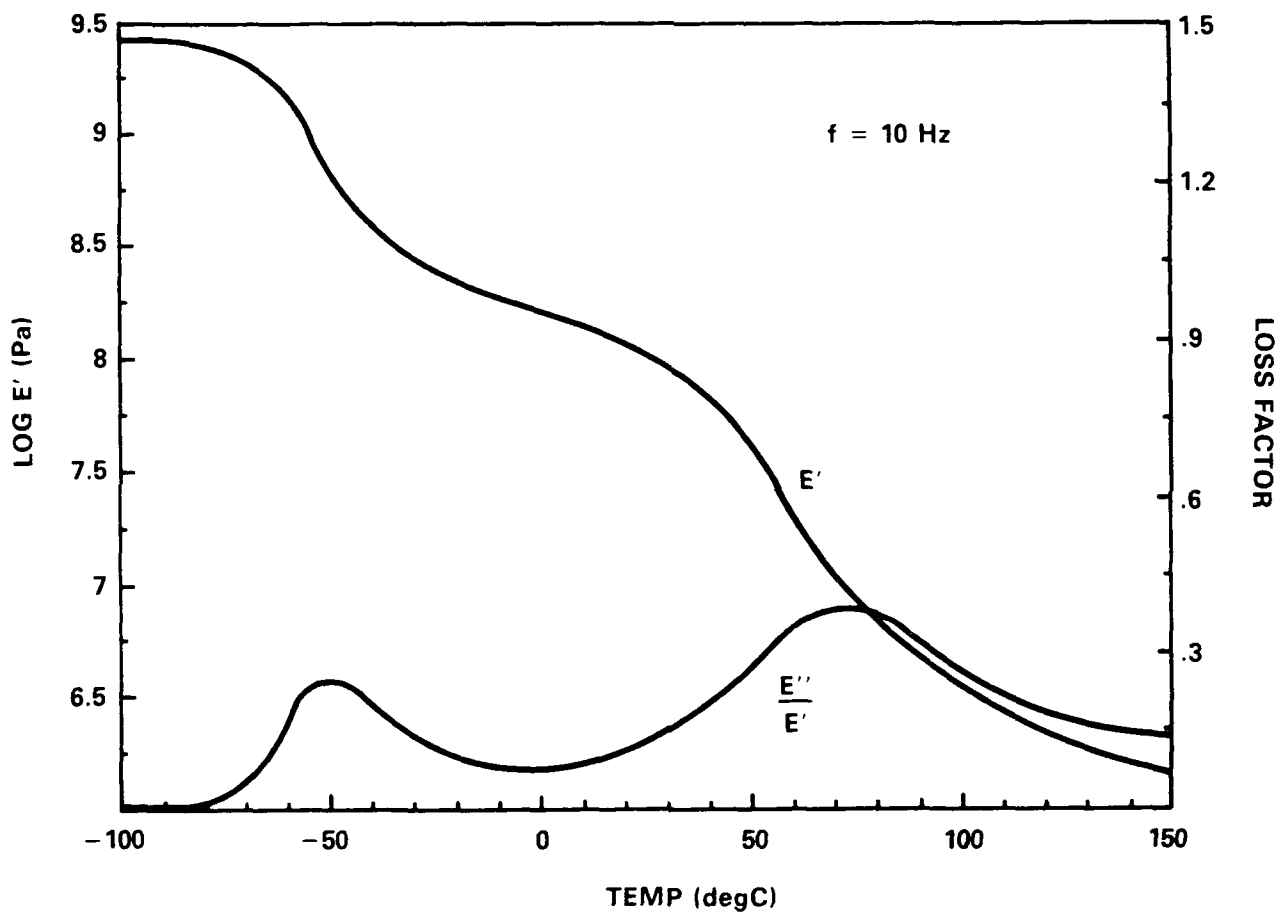
FIGURE 4. YOUNG'S MODULUS AND LOSS FACTOR FOR CONAP
EN-4 AT 10 HZ AS A FUNCTION OF TEMPERATURE
MEASURED BY THE DMTA
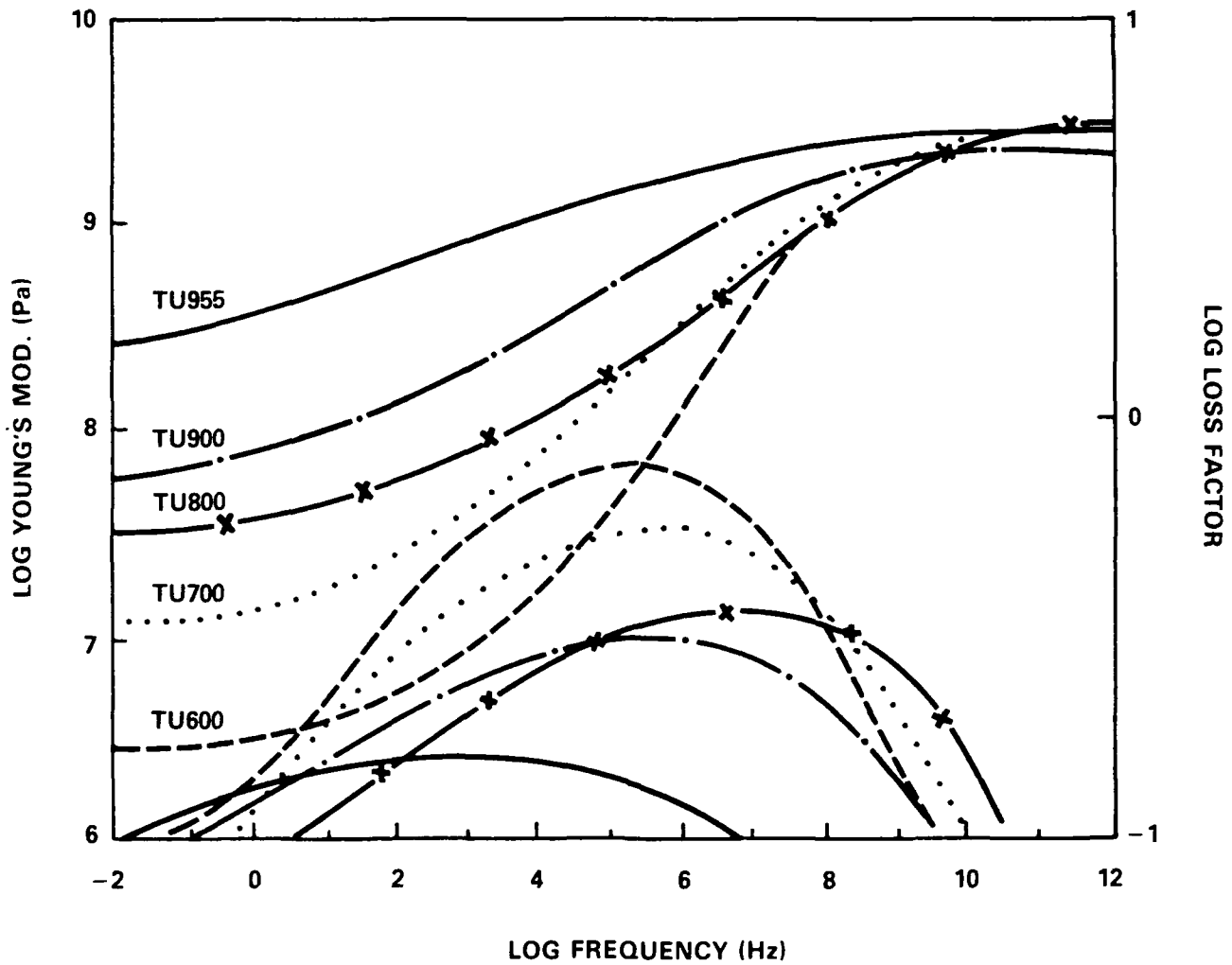
FIGURE 5. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND
LOSS FACTOR FOR A SERIES OF URETHANE POLYMERS
AS A FUNCTION OF FREQUENCY AT 10°C
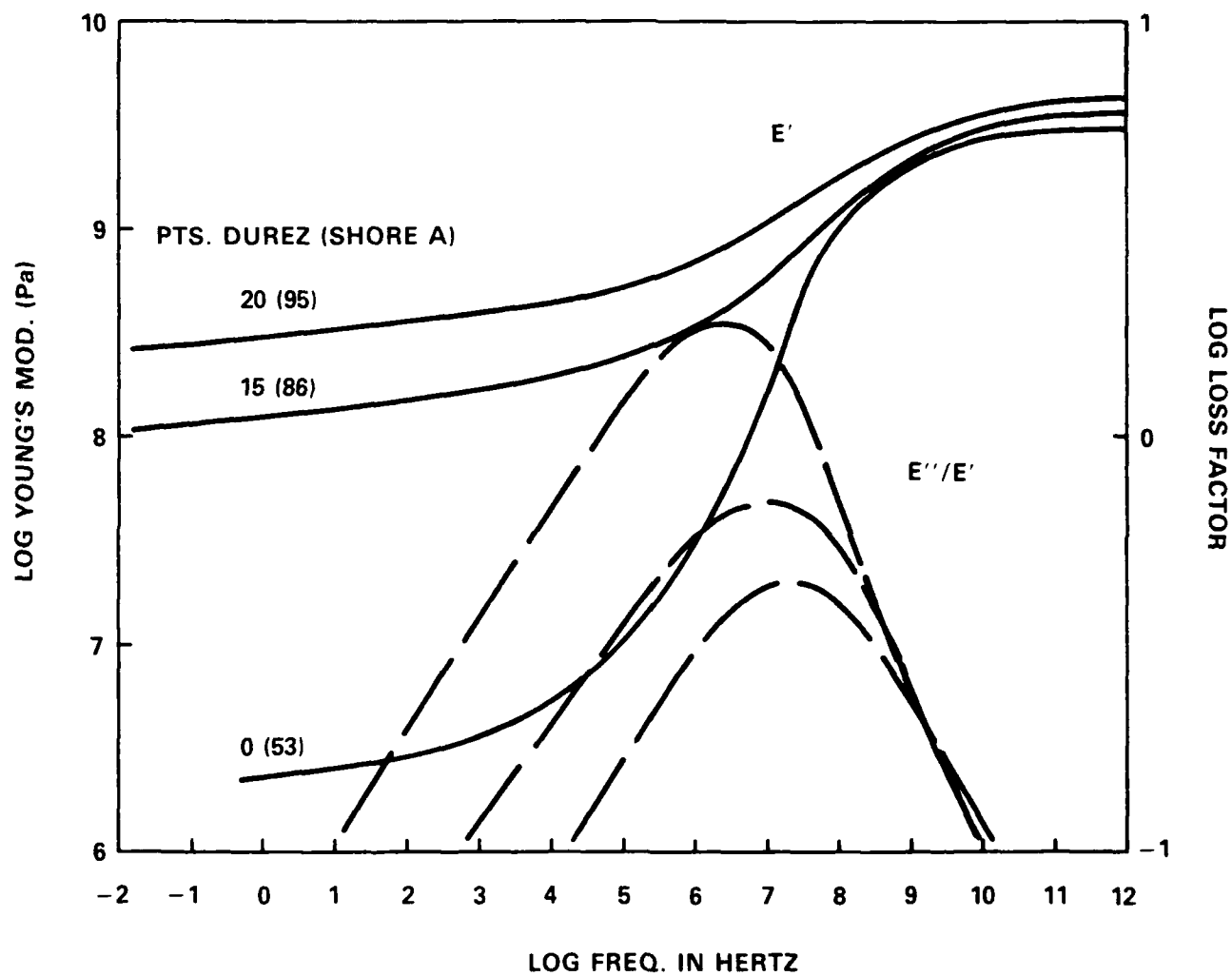
FIGURE 6. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND
LOSS FACTOR FOR A SERIES OF NATURAL RUBBER
COMPOUNDS AS A FUNCTION OF FREQUENCY AT 10°C
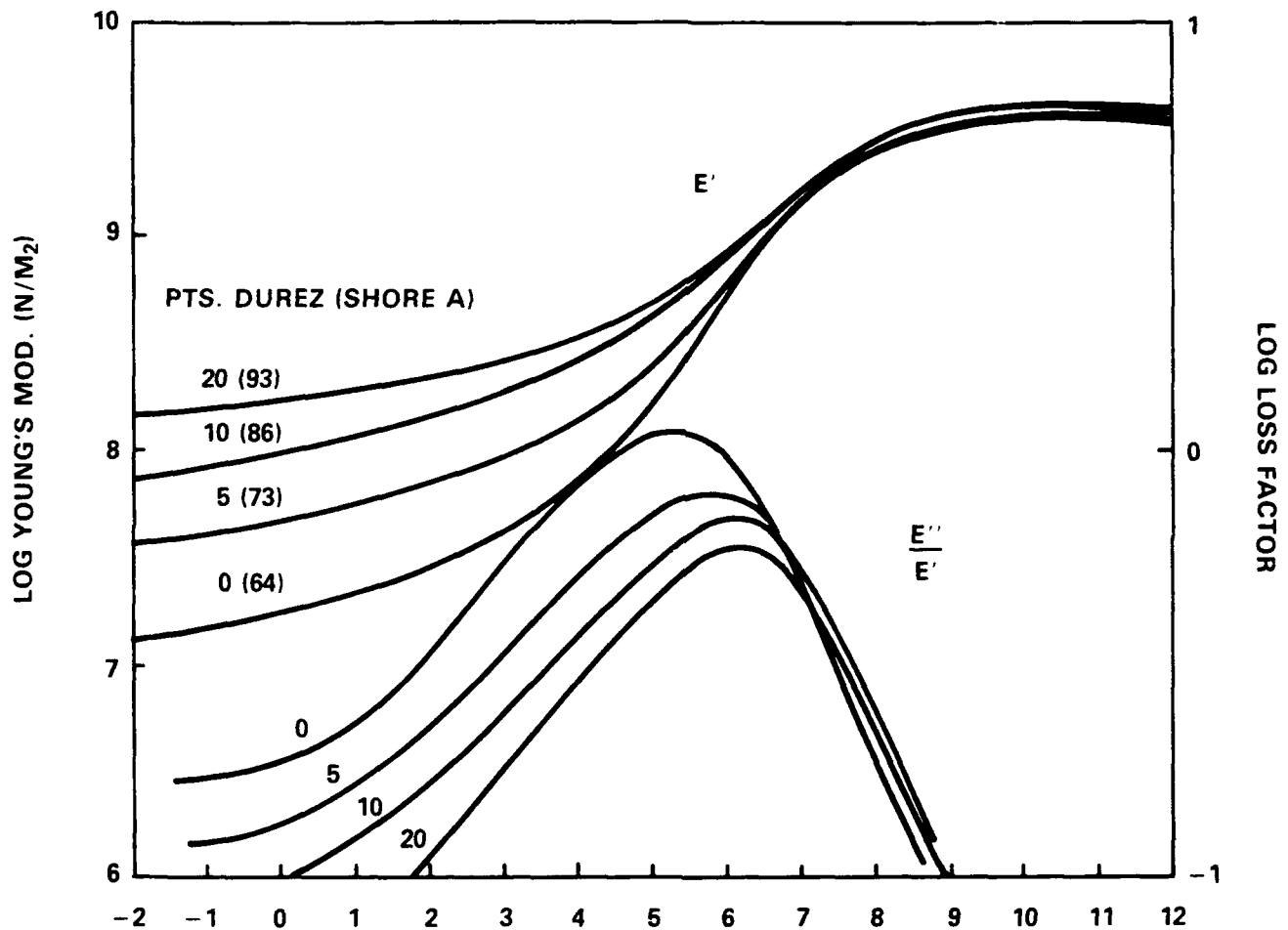
FIGURE 7. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND
LOSS FACTOR FOR A SERIES OF NEOPRENE RUBBER
COMPOUNDS AS A FUNCTION OF FREQUENCY AT 10°C

13
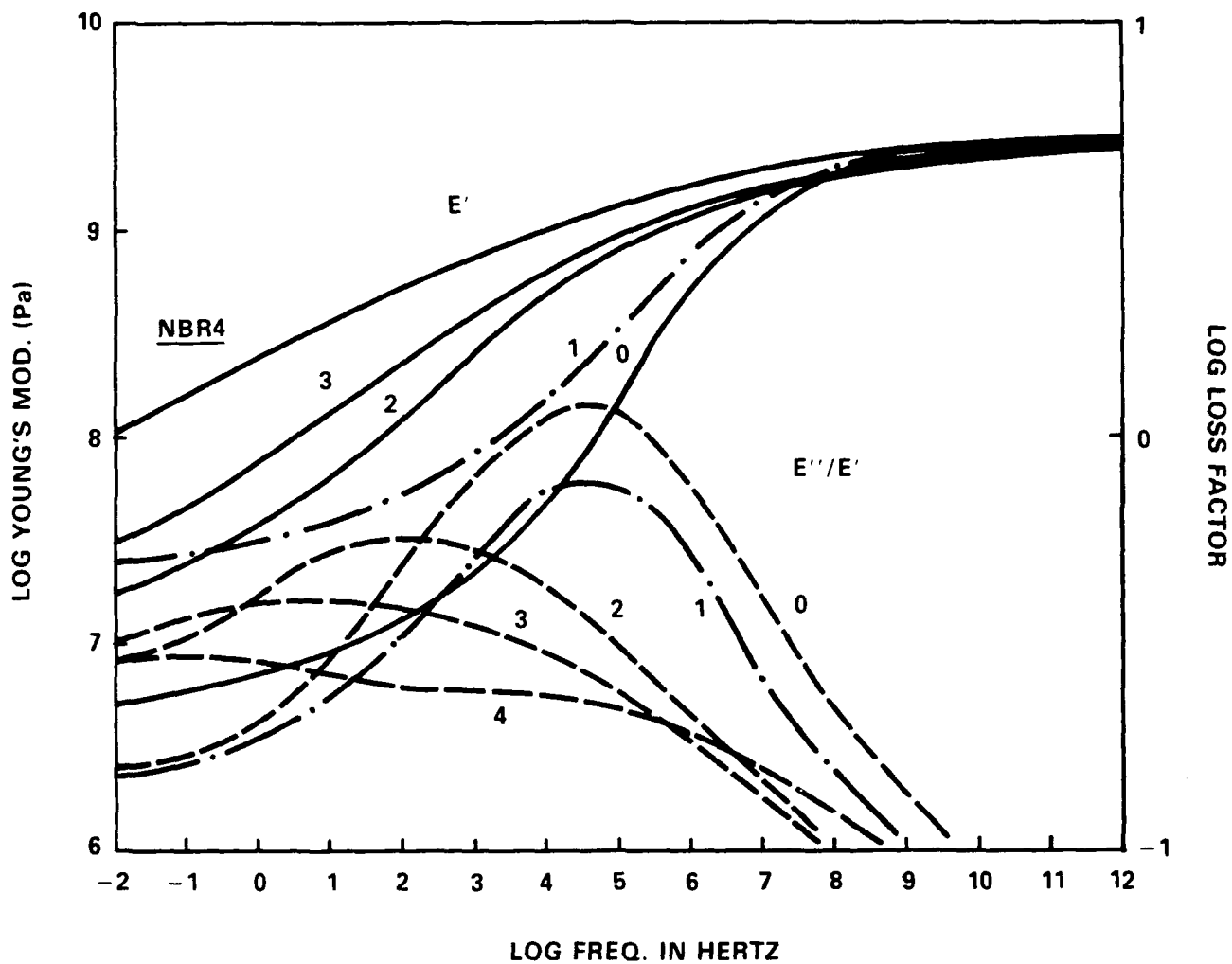
FIGURE 8. THE WLF SHIFTED DYNAMIC YOUNG'S MODULUS AND LOSS
FACTOR FOR A SERIES OF NITRILE RUBBER COMPOUNDS
AS A FUNCTION OF FREQUENCY AT 10°C

TABLE 1. RUBBER FORMULATIONS USED IN THIS STUDY

Compound #	NR-1, 2, 3	NBR-0, 1, 2, 3, 4	1, 2, 3, 4
Natural SMR-20	100	---	---
Paracril BLT	---	100	---
Neoprene NPR6398	---	---	100
Black N330	---	---	---
Black N660	20, 65, 65	0, 60, 40, 40, 40	---
Black N990	---	---	100
Protox 166	5	5	---
Steric acid	3	3	1
Dicup 40C	---	4.25	---
Red lead 95%	---	---	15
Sulfur	2.2	---	---
Altax	2.5	---	1.5
Circo light oil	3	---	---
Octamine	2	1	2
DOP	---	15, 0, 0, 0, 0	---
Califux	---	0, 5, 5, 5, 5	---
Age Rite Resin D	---	0.5	---
TE-70	---	---	2
Durez 12687	0, 15, 20	0, 0, 20, 30, 40	0, 5, 10, 20

TABLE 2. FRACTIONAL DERIVATIVE CONSTANTS FOR NITRILE
COMPOUNDS

Compound #	LOG Er (Pa)	LOG Eg (Pa)	Fo (MHz)	a
NBR-0	6.85	9.40	2.3	.58
NBR-1	7.48	9.40	10.0	.42
NBR-2	6.60	9.40	0.17	.33
NBR-3	7.36	9.40	0.10	.30
NBR-4	7.95	9.48	1.0	.23

REFERENCES

1. Ferry, J., <u>Viscoelastic Properties of Polymers</u>, John Wiley and Sons, New York, NY, 1970.

2. Rogers, L., "Operators and Fractional Derivatives for Viscoelastic Constitutive Equations," <u>J. Rheology</u>, Vol. 27, No. 4, 1983, pp. 351-372.

3. Polymer Laboratories, Inc., 160 Old Farm Road, Amherst, MA 01002.

4. Madigosky, W. M., and Lee, G. F., "Improved Resonance Technique for Materials Characterization," <u>J. Acoust. Soc. Am.</u>, Vol. 73, 1983, pp. 1374-1377.

DISTRIBUTION

	Copies		Copies
Commander Naval Sea Systems Command Attn: Code 55W (J. Keggler) Washington, DC 20362	1	Commander Naval Coastal Systems Center Attn: Technical Library Panama City, FL 32407	1
Commanding Officer Naval Research Laboratory Attn: Code 5135 (R. Corsaro) 4555 Overlook Avenue, S.W. Washington, DC 20375	1	Commanding Officer Mare Island Naval Shipyard Attn: Code 134.04 (B. Jan) Ship Silencing Branch Vallejo, CA 94592	1

Commander
Naval Underwater Systems Center
Attn: Code 332 (R. Radlinksi)
      Code 2133 (Roger Maple)
      Code 2141 (S. Austin)
      Technical Library
New London Laboratory
New London, CT 06320
	1
	1
	1
	1
		Commanding Officer
		Underwater Sound Reference
		  Division
		Attn: Code 5995
		      (Dr. C. Thompson)
		      (Dr. R. N. Capps)
		      Technical Library
		Orlando, FL 32806
			
			1
			1
			1

Commander
David Taylor Research Center
Attn: Code 1908
      (S. McKeon)
      (J. Dlubac)
      Technical Library
Bethesda, MD 20084
	
	1
	1
	1
		Office of the Chief of
		  Naval Research
		Attn: ONR-1131 (R. Pohanka)
		      ONR-1132-SM
		      (Y. Rajapekse)
		      (Albert Tucker)
		      (Stephen Newfield)
		      ONT-225 (W. T. Messick)
			1
			
			1
			1
			1
			1

Commander
David Taylor Ship Research and
  Development Center
Attn: Code 2842 (A. Santiago)
      Technical Library
Annapolis Laboratory
Annapolis, MD 21402
	
	1
	1
		800 No. Quincy Street
		Arlington, VA 22217-5000
		
		Wright Laboratory
		Attn: WL/FIBG
		      (Dr. D. Jones)
		      (Dr. L. Rogers)
		Wright-Patterson AFB
		OH 45433
			
			1
			1

Commander Naval Ocean Systems Center Attn: Library San Diego, CA 92152	1		

DISTRIBUTION (Cont.)

	Copies		Copies
Air Force Institute of Technology Attn: AFIT/ENY (Lt.Col. R. Bagely) Wright-Patterson AFB OH 45433	1	Materials Research Center Lehigh University Attn: Dr. L. Sperling Whitaker Laboratory #5 Bethlehem, PA 18015	1
CSA Engineering, Inc. Attn: B. R. Allen 560 San Antonio Road, Suite 101 Palo Alto, CA 94306	1	Library of Congress Attn: Gift & Exchange Division Washington, DC 20540	4
Bolt, Beranek, and Newman, Inc. Attn: Dr. E. M. Kerwin 10 Moulton Street Cambridge, MA 02238	1	Defense Technical Information Center Cameron Station Alexandria, VA 22304-6145	12

Internal Distribution:

	Copies
E231	3
E232	2
E35	1
R	1
R30	2
R31	1
R31 (W. Madigosky)	4
R31 (K. Scharnhorst)	1

	Copies
E. I. DuPont Attn: R. W. Megill 6 Polaris Drive Newark, DE 19711	1
Anatol Corporation Attn: A. D. Nashif 10895 Indeco Drive Cincinnati, OH 45241	1
Vector Research Company, Inc. Attn: Dr. W. Reader 6903 Rockledge Drive, Suite 1200 Bethesda, MD 20817	1
General Dynamics/Eastern Point Road Electric Boat Division Attn: J. Krause Groton, CT 06340	1
The Pennsylvania State University Applied Research Laboratory Attn: Library P.O. Box 30 State College, PA 16804	1

REPORT DOCUMENTATION PAGE

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE January 1991	3. REPORT TYPE AND DATES COVERED FY 1988-1989

4. TITLE AND SUBTITLE
Dynamic Mechanical Materials Characterization and Analysis Using a Dynamic Mechanical Thermal Analyzer

5. FUNDING NUMBERS

6. AUTHOR(S)
Dr. Walter M. Madigosky

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Naval Surface Warfare Center
White Oak Laboratory (Code R31)
10901 New Hampshire Avenue
Silver Spring, MD 20903-5000

8. PERFORMING ORGANIZATION REPORT NUMBER

NAVSWC TR 91-90

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

David Taylor Research Center
Attn: Code 1908
Bethesda, MD 20084

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Material characterization is essential both for design use and control. Specifically, materials designed for vibration damping and noise suppression precise values of the modulus and loss factor over a wide range of frequency and temperature. Until now, apparatus for making such measurements had very limited frequency and temperature ranges, were of questionable accuracy, and were usually very time consuming to operate. This report presents data as a function of temperature, from the glassy to the rubbery region, measured on a Polymer Laboratories' Dynamic Mechanical Thermal Analyzer (DMTA) and the same data transformed into the frequency domain using the Williams-Landel-Ferry (WLF) time-temperature shift equation. Data were collected on a number of natural, neoprene, nitrile, and polyurethane materials with different levels of reinforcement filler. The data clearly show the effect of polymer selection, filler type, and loading on the dynamic properties. Finally, the complex moduli may be analyzed in terms of the fractional operator model which describes both the storage and loss moduli with a single complex equation. The results suggest an analytic method for optimizing the dynamic response through detailed material selection.

14. SUBJECT TERMS
Dynamic Mechanical Thermal Analyzer (DMTA)
neoprene   nitrile   polymer
polyurethane

15. NUMBER OF PAGES
27

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT SAR

GENERAL INSTRUCTIONS FOR COMPLETING SF 298

The Report Documentation Page (RDP) is used in announcing and cataloging reports. It is important that this information be consistent with the rest of the report, particularly the cover and its title page. Instructions for filling in each block of the form follow. It is important to *stay within the lines* to meet *optical scanning requirements*.

Block 1. Agency Use Only *(Leave blank)*.

Block 2. Report Date. Full publication date including day, month, and year, if available (e.g. 1 Jan 88). Must cite at least the year.

Block 3. Type of Report and Dates Covered. State whether report is interim, final, etc. If applicable, enter inclusive report dates (e.g. 10 Jun 87 - 30 Jun 88).

Block 4. Title and Subtitle. A title is taken from the part of the report that provides the most meaningful and complete information. When a report is pre- pared in more than one volume, repeat the primary title, add volume number, and include subtitle for the specific volume. On classified documents enter the title classification in parentheses.

Block 5. Funding Numbers. To include contract and grant numbers; may include program element number(s), project number(s), task number(s), and work unit number(s). Use the following labels:

C	-	Contract	PR	- Project
G	-	Grant	TA	- Task
PE	-	Program Element	WU	- Work Unit Accession No.

BLOCK 6. Author(s). Name(s) of person(s) responsible for writing the report, performing the research, or credited with the content of the report. If editor or compiler, this should follow the name(s).

Block 7. Performing Organization Name(s) and Address(es). Self-explanatory.

Block 8. Performing Organization Report Number. Enter the unique alphanumeric report number(s) assigned by the organization performing the report.

Block 9. Sponsoring/Monitoring Agency Name(s) and Address(es). Self-explanatory.

Block 10. Sponsoring/Monitoring Agency Report Number. *(If Known)*

Block 11. Supplementary Notes. Enter information not included elsewhere such as: Prepared in coop- eration with...; Trans. of...; To be published in.... When a report is revised, include a statement whether the new report supersedes or supplements the older report.

Block 12a. Distribution/Availability Statement. Denotes public availability or limitations. Cite any availability to the public. Enter additional limitations or special markings in all capitals (e.g. NOFORN, REL, ITAR).

DOD	-	See DoDD 5230.24, "Distribution Statements on Technical Documents."
DOE	-	See authorities.
NASA	-	See Handbook NHB 2200.2
NTIS	-	Leave blank.

Block 12b. Distribution Code.

DOD	-	Leave blank.
DOE	-	Enter DOE distribution categories from the Standard Distribution for Unclassified Scientific and Technical Reports.
NASA	-	Leave blank.
NTIS	-	Leave blank.

Block 13. Abstract. Include a brief *(Maximum 200 words)* factual summary of the most significant information contained in the report.

Block 14. Subject Terms. Keywords or phrases identifying major subjects in the report.

Block 15. Number of Pages. Enter the total number of pages.

Block 16. Price Code. Enter appropriate price code *(NTIS only)*

Blocks 17.-19. Security Classifications. Self- explanatory. Enter U.S. Security Classification in accordance with U.S. Security Regulations (i.e., UNCLASSIFIED). If form contains classified information, stamp classification on the top and bottom of the page.

Block 20. Limitation of Abstract. This block must be completed to assign a limitation to the abstract. Enter either UL (unlimited) or SAR (same as report). An entry in this block is necessary if the abstract is to be limited. If blank, the abstract is assumed to be unlimited.